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IS 6889 (2000): Chemical Analysis of Silver-tin Dental Amalgam Alloy - Method [MTD 10: Precious Metals]



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दांतो के लिए रजत-टिन अमलगम
मिश्रधातु का रासायनिक विश्लेषण — पद्धति
(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF SILVER-TIN DENTAL
AMALGAM ALLOY — METHOD
(*First Revision*)

ICS 11.060.10:71.040.40

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Silver-tin dental amalgam alloy the composition of which is given in IS 4704:1985 'Silver-tin dental amalgam alloy (*first revision*)' is used in dentistry. This alloy contains silver-tin, copper and zinc. IS 6889 was first published in 1973 which prescribes the method of chemical analysis of dental amalgam alloy. This standard is being revised on the basis of experience gained during the past.

During this revision following changes have been made:

- a) The determination of tin by gravimetric has been updated.
- b) The determination of silver by the gravimetric method has been updated and an alternate method for determination of silver by 'Potentiometric titration method' has been included.
- c) The determination of copper by 'Electrolytic method' has been updated and the Polarographic method has been included.
- d) The determination of zinc by the photometric has been replaced by the method for simultaneous determination of copper and zinc by polarographic method.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF SILVER-TIN DENTAL AMALGAM ALLOY—METHOD

(First Revision)

1 SCOPE

This standard describes the methods for determination of silver, tin, copper and zinc in the dental amalgam alloy in the ranges given below:

<i>Element</i>	<i>Range (Percent by Mass)</i>
Silver	66.7-74.5
Tin	25.8-27.0
Copper	0.0-6.0
Zinc	0.1-1.9

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
266 : 1977	Sulphuric acid (<i>second revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
6882 : 1973	Platinum electrode

3 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070) shall be employed for the tests.

4 SAMPLING

The sample is in the form of fillings or shavings. If in the form of shavings the sample shall be cut into small pieces and shall then be gone over by a magnet to remove extraneous iron. It shall be thoroughly mixed, quartered and then drawn.

5 DETERMINATION OF TIN BY THE GRAVIMETRIC METHOD

5.1 Outline of the Method

Tin is separated by dissolving the sample in nitric acid when a precipitate of meta-stannic acid is formed. The precipitate is filtered, washed, dried,

ignited and weighed. The weight of stannic oxide (SnO_2) is determined by volatilization with ammonium iodide.

5.2 Reagents

5.2.1 Nitric Acid — r.d. = 1.42 (conforming to IS 264).

5.2.2 Dilute Nitric Acid — 1:99 (v/v).

5.2.3 Ammonium Iodide — Solid.

5.3 Procedure

5.3.1 Weigh accurately 0.50 g of the sample into a 200 ml beaker and cover with a watch glass. Add a mixture of 15 ml of nitric acid and 10 ml of water by removing the watch glass and replace it immediately. When the vigorous action is over, evaporate the solution for a period of at least 1 h on a steam-bath to a syrupy consistency. Dilute to about 150 ml with dilute nitric acid (1:99) and heat on a water-bath for 30 min.

5.3.2 Allow the precipitate to settle overnight. Transfer the clear solution over a medium textured filter paper containing a little ashless filter pulp, retaining the precipitate in the beaker. Wash the residue in the beaker thrice with 10 ml portions of the hot dilute nitric acid (1:99) and transfer the residue to the filter paper. Wash the residue further at least five times with hot dilute nitric acid (1:99).

NOTE — Every precautions should be taken to avoid contamination by chloride in order to prevent the precipitation of silver chloride.

5.3.3 Place the filter paper along with the residue in a weighed silica crucible. Dry carefully and char the filter paper at a low temperature and finally ignite the residue for 30 min in a muffle furnace at about 800°C.

5.3.4 Moisten the precipitate with two drops of nitric acid (dilute). Evaporate and ignite again. Allow to cool in a desiccator and weigh. Repeat the ignition till constant mass is obtained.

5.3.5 Mix the precipitate with 15 times its mass of pure ammonium iodide and heat in a muffle furnace at 425 to 475°C till no further fumes are evolved and tin volatilizes completely. Treat the residue with 2 to 3 ml of nitric acid, evaporate completely, ignite

at 800°C and weigh the residual metallic oxide. The loss in weight gives the mass of stannic oxide.

5.3.6 Dissolve the residue in nitric acid and mix it with the main filtrate. Make upto 250 ml and preserve the filtrate for the determination of silver, copper and zinc.

NOTE — If any residue is left, it should be checked for silver as per the method given in 6.

5.3.7 Carry out a black determination using the same amount of ammonium iodide used in the procedure.

5.4 Calculation

$$\text{Tin, percent by mass} = \frac{(A - B) \times 78.77}{C}$$

where

- A = mass in g of stannic oxide,
 B = mass in g of residue obtained from the blank, and
 C = mass in g of the sample taken.

6 DETERMINATION OF SILVER BY THE GRAVIMETRIC METHOD (REFREE METHOD)

6.1 Outline of the Method

Silver is precipitate from the solution as silver chloride with the help of hydrochloric acid. The precipitate is filtered, washed, dried and weighed.

6.2 Reagents

6.2.1 Dilute Hydrochloric Acid — 1:4 (v/v).

6.2.2 Dilute Nitric Acid — 1:99 (v/v).

6.3 Procedure

6.3.1 Take the filtrate preserve under 5.3.6 and add drop by drop dilute hydrochloric acid (1:4) with constant stirring till the precipitation is complete. Add one ml more of acid. Heat and allow the precipitate to settle down, at a place away from the sunlight. Test the supernatant liquid with one or two drops of dilute hydrochloric acid to make sure that the precipitation is complete. Allow the precipitate to stand in dark for 2 h or preferably overnight.

6.3.2 Filter through a weighed sintered glass crucible (porosity 4) and wash several times with dilute nitric acid till the washings are free from chlorides. Dry the crucible and precipitate at 120 to 130°C for about 1 h. Cool in a dessicator to room temperature and weigh.

6.3.3 Reserve the filtrate for the determination of copper.

6.4 Calculation

$$\text{Silver, percent by mass} = \frac{A}{B} \times 75.26$$

where

A = mass in g of silver chloride, and

B = mass in g of sample taken.

7 DETERMINATION OF SILVER BY POTENTIOMETRIC TITRATION METHOD (ALTERNATE METHOD)

7.1 Outline of the Method

The filtrate obtained from the precipitation of tin is taken up for silver analysis. Potentiometric titration of 25 ml of the filtrate is carried out against potassium chloride employing either a Ag/Pt bi-metallic electrode system or Ag/saturated mercurous sulphate reference electrode.

7.2 Reagents

7.2.1 Stock Silver Nitrate Solution (0.1 M)

Keep solid silver nitrate in an oven at 160°C in a dust free atmosphere for 3 h and then cool in the dessicator. Weigh accurately 16.989 g of it and dissolve in deionized water. Dilute the solution to 1 litre and store in an amber coloured bottle.

7.2.1.1 Standard Silver Nitrate Solution (0.01 M)

Transfer 25 ml of stock silver nitrate (7.2.1) to a 250 ml flask and make up the solution to the mark with deionized water.

7.2.2 Stock Potassium Chloride Solution (0.1 M)

Dry potassium chloride at 130°C in an oven for 3-4 h and cool in a dessicator. Weigh 7.450 g of it and dissolve in deionized water and make up to 1 litre.

7.2.2.1 Standard potassium chloride solution (0.01 M)

Transfer 25 ml of stock potassium chloride solution (7.2.2) to a 250 ml flask and make up to the mark with deionized water.

7.2.3 Dilute Sulphuric Acid — 1:4 (v/v).

7.2.4 Dilute Nitric Acid — 1:4 (v/v).

7.3 Apparatus

7.3.1 pH meter, having the facility of measuring both pH and potentials (see Note). Silver electrode is to be connected to the joint where glass electrode is to be connected and platinum or mercurous sulphate electrode is connected to the joint where calomel electrode is connected.

NOTE — If in the potential mode, two ranges of potential, namely 0-1 200 mV and 0-200 mV are provided, the later may be chosen.

7.3.2 Electrodes

7.3.2.1 Silver wire electrode (diameter 1.0 mm)

It is prepared by sealing the wire in a soft glass tube of internal diameter 1 cm and length of 8-10 cm in such a way that 2 cm wire is put out of the glass tube, and a 0.5 cm remains in the glass tube. Four mercury in the tube for electrical contact.

7.3.2.2 Platinum wire electrode

Prepared in the same way as given in 7.3.2.1 except pyrex tube used in place of soft glass tube.

7.3.2.3 Saturated mercurous sulphate electrode

Take a glass tube of length 6 cm and 2 cm diameter. Fuse a platinum wire at its bottom in such a way that 0.5 cm wire is inside the tube and 0.5 cm wire is outside the tube (see Note 1). Pour mercury inside the glass tube, till the platinum wire inside the tube is submerged and then pour mercurous sulphate paste till it attains a height of 0.5 cm. Provide the glass tube with a side tube having 6 mm diameter and attached near the top that is nearly at a height of 4 cm from the bottom. The side is bent at right angle and the end of the tube is plugged with a ceramic plug or filter paper. Pour saturated potassium sulphate solution over mercurous sulphate past (see Note 2) till the side tube is filled up. Cap the main glass tube, preferably with plastic cap.

The potential of saturated mercury/mercurous sulphate electrode is 0.64 V vs Normal Hydrogen electrode (NHE).

NOTES

- 1 The wire outside the tube is used for electrical connections.
- 2 Mercurous sulphate paste is prepared in an agate mortar by mixing a little pure mercury, solid mercurous sulphate and very small amounts of saturated potassium sulphate solution.

7.4 Electrode Cleaning (Pretreatment)

7.4.1 Clean silver electrode by keeping it in dilute nitric acid (1:4) for 5 min and wash it repeatedly with deionized water.

7.4.2 Clean platinum wire by making it cathode in a solution of dilute sulphuric acid (1:4). Take dry cell of 1.5 V and connect the platinum wire electrode to the cathode. Join another platinum wire electrode to the anode of the dry cell. Dip both the electrodes in a solution of dilute sulphuric acid (1:4) taken in a 50 ml beaker and carry out the electrolysis for 5 min.

Change the polarities of electrodes and carry out electrolysis for 1 min. Change the polarity again to their original status and carry out electrolysis for 5 min. Then wash platinum wire electrodes (cathode)

thoroughly with deionized water and take up for titration work.

Clean both the electrodes before the start of each titration.

7.5 Procedure

7.5.1 Pipette out 25 ml of the solution preserved under 5.3.6 into a 100 ml beaker, fitted with a rubber bung having three holes (two for inserting the two electrodes and one for inserting a burette). Place a small magnetic stirring bar in the beaker and stir the solution by means of a small motor.

NOTES

- 1 The stirring should not be turbulent.
- 2 Two electrode system may be used to follow the change of potential with titrant addition. Take potassium chloride solution (0.01 M) in the burette.

7.5.2 Bi-metallic Electrode System

Potentials are recorded employing the bi-metallic electrode system Ag/Pt and a graph of potential vs volume in ml of potassium chloride solution (0.01 M) added, is plotted and equivalence point is obtained from the curve.

7.5.3 Ag-Hg ($Hg_2 SO_4$) Electrode System

Titration is carried out exactly in the same way as detailed in 7.5.2. except that platinum electrode is replaced by mercurous sulphate electrode. In this titration, the graph of potential vs volume in ml of potassium chloride solution (0.01 M) is 'S' shaped and the end point is computed from the potentiometric height of the curve. The volume in ml of titrant obtained for one half of the total potentiometric curve height corresponds to the end point.

NOTES

- 1 While carrying out the titration, the increment of titrant addition should be very small that is 0.1 ml near the end point irrespective of the methods followed.
- 2 To check the method and pH meter, 25.0 ml of silver nitrate (0.01 M) in the beaker is titrated against potassium chloride (0.01 M) in the burette. The expected and experimental end points should tally within 0.1 ml.

7.5.4 Calculation

1.0 ml of 0.01 M potassium chlorride = 0.001 08 g/ml of silver
 V ml of 0.01 M potassium chloride required for end point =

$$V \times 0.001\ 08$$

Silver content in the original 250 ml of filtrate =

$$V \times 0.001\ 08 \times 10\ \text{g of silver}$$

If the sample weight is W gram then silver percentage by mass :

$$= \frac{V \times 1.08}{W}$$

where

V = volume in ml of 0.01M KCl required for the end point, and

W = mass in g of sample taken.

8 DETERMINATION OF COPPER BY THE ELECTROLYTIC METHOD

8.1 Outline of the Method

Copper is deposited electrolytically on a platinum cathode and weighed.

8.2 Apparatus

Any of the platinum electrodes specified in IS 6882 may be used.

8.3 Reagents

8.3.1 Sulphuric Acid — r.d. = 1.84 (conforming to IS 266).

8.3.2 Nitric Acid — see 5.2.1.

8.3.3 Urea — Solid.

8.3.4 Sulphamic Acid — Solid.

8.3.5 Rectified Spirit — 95 percent (free from solid matter).

8.4 Procedure

8.4.1 To the filtrate reserved under 6.3.3 add 5 ml of sulphuric acid and evaporate to dense white fumes. Cool, dilute with 50 ml of water and boil. Transfer to a 400 ml beaker, add 2 ml of nitric acid and dilute to about 150 ml with water. Add 0.5 g of urea or 0.1 g of sulphamic acid, boil and cool. Dip the electrodes into the solution. Cover with watch glass.

8.4.2 Electrolyze at a current density of $5A/dm^2$, using cathode and rotating agitator, for about an hour. When the solution becomes colourless, wash down the cover glass, electrodes and sides of the beaker with water, raising the level of the liquid slightly and continue the electrolysis until no copper is deposited on the newly exposed surface of the cathode.

8.4.3 When the deposition is complete, discontinue the electrolysis, and raise the cathode gradually as it is thoroughly washed with water, collecting the washings in the beaker. Dip the cathode in two successive baths of rectified spirit. Dry for 3 to 5 min at 105° to $110^\circ C$. Cool in a dessicator to room temperature and weigh the deposit as metallic copper.

8.5 Calculation

Copper, percent by mass = $\frac{A}{B} \times 100$

where

A = mass in g of copper, and

B = mass in g of sample taken.

9 SIMULTANEOUS DETERMINATION OF COPPER AND ZINC BY POLAROGRAPHIC METHOD

9.1 Outline of the Method

Copper and zinc in the range below 4 percent are simultaneously determined by the Dropping Mercury Electrode (DME) employing acetate buffer of 4.63 pH by polarography.

9.2 Apparatus

9.2.1 Polarograph

A manual or a recording polarograph with a three electrode system may be used. DME will act as a working electrode, platinum wire electrode (1.5 cm long) as auxiliary electrode and Saturated Calomel Electrode (SCE) as a reference electrode.

NOTES

1 If a manual polarograph is to be used, then only two electrodes namely DME and SCE are employed.

2 For a recording polarograph, the voltage scan rate is to be kept constant preferably at 5-6 mV/s.

9.3 Reagents

9.3.1 Mercury — Double distilled.

9.3.2 Nitric Acid — r.d. = 1.42 (conforming to IS 264).

9.3.3 Dilute Nitric Acid — (1:1) (v/v).

9.3.4 Acetate Buffer Solution (pH = 4.63)

Add 50 ml of 0.2 M acetic acid to 50 ml of sodium acetate 0.2 M.

9.3.5 Standard Copper Solution (100 $\mu g/ml$)

Dissolve 0.1 g of copper (electrolytic purity grade) in 5.0 ml of dilute nitric acid (1:1) in a 50 ml beaker. Cover with a watch glass. Evaporate the solution to dryness on water-bath. Wash watch glass and beaker with double distilled water and transfer the contents to 1 litre volumetric flask after addition of 1.0 ml of nitric acid and make up to mark with double distilled water.

9.3.5.1 Standard Copper Solution (10 $\mu g/ml$)

Transfer 10 ml of standard copper solution (8.3.5) to 100 ml volumetric flask and make up to the mark with water.

9.3.6 Standard Zinc Solution (100 $\mu g/ml$)

Dissolve 0.1 g of pure zinc in 5 to 10 ml dilute hydrochloric acid, cool and dilute to 1 litre in a volumetric flask.

9.3.6.1 Standard Zinc Solution (10 µg/ml)

Transfer 10 ml of standard zinc solution (9.3.6) to 100 ml volumetric flask and make upto the mark with water.

9.3.7 Gelatin Solution (0.1 percent)

Weigh 0.1 g of pure gelatin and dissolve in hot water till a clear solution is obtained. Transfer it to a 100 ml volumetric flask and make upto the mark with warm water. Allow to cool for some time and again make up the solution to the mark with cold water.

9.3.8 Nitrogen Gas

Pure nitrogen gas free from traces of oxygen is required to deoxygenate the solution.

9.3.9 Polarographic Cell

A 100 ml pyrex beaker fitted with a rubber or plastic cap having five holes may be used. Working, auxiliary and reference electrode are to be inserted in the three holes. A glass tube jointed to the nitrogen cylinder by means of polythene tube is inserted through the fourth hole and the fifth hole is meant for exit of nitrogen gas.

9.4 Procedure

Pipette out 25 ml of the filtrate obtained after precipitation of tin (5.3.6) to a 250 ml flask and make upto the mark with double distilled water. From this solution pipette 2.5 ml of the solution to a 25 ml volumetric flask. Add 12.5 ml of acetate buffer and 2.5 ml gelatin solution and then make upto the mark. Transfer the content to the 100 ml pyrex beaker which serves as the cell and pass pure nitrogen through the solution for 15 min. After that pull up the nitrogen bubbling tube above the solution and let nitrogen pass above the cell solution during the experiment.

Adjust the initial voltage to + 0.05 V and apply the potential till hydrogen discharge wave is obtained. Measure the height of the two waves.

NOTE — Copper reduction wave is obtained between + 0.05 to -0.25 V and zinc wave is obtained between -0.9 to -1.3V.

9.5 Calibration

Calibration is preferably done by spiking. To the polarographic cell solution, add 0.1 ml or more of the standard copper and zinc solutions (100 µg/ml) so that heights of the waves are nearly double. Calculate the total height, obtained after addition of the standard solutions and from it subtract the height obtained for the sample alone. The difference will give the equivalence between the µg of copper or zinc added and the currents obtained.

NOTE — If copper and zinc are present in traces the addition should be done from 10 µg/ml standard solution.

9.6 Calculation

If Y_1 µA is the current obtained for 25 ml of sample solution after adding X µg of standard (volume of standard solution added multiplied by concentration of the standard solution in µg/ml and Y_2 µA is the current obtained for the sample solution alone, then, $(Y_1 - Y_2)$ µA is the current for X µg of the standard in 25 ml of sample solution (presuming that the volume of standard solution is negligible as compared to sample solution) and therefore Y_2

corresponds to $\frac{X \times Y_2}{Y_1 - Y_2}$ µg of analyte in

sample solution and therefore

Percentage by mass of

$$\text{zinc/copper} = \frac{10\,000 \times 10^2 \times 10^{-6}}{\frac{A}{W} \times 0.1}$$

where

A = mass of zinc/copper in µg, and

W = mass of sample taken in g.

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